Institute for Reference Materials and Measurements

ERM European Reference Materials

CERTIFICATION REPORT

Certification of Charpy V-notch reference test pieces of 150 J nominal absorbed energy

Certified Reference Material ERM®-FA415t

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European Commission
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Contact information
Reference materials sales
Retieseweg 111
B-2440 Geel, Belgium
E-mail: jrc-irmm-rm-sales@ec.europa.eu
Tel.: +32 (0)14 571 705
Fax: +32 (0)14 590 406

http://irmm.jrc.ec.europa.eu/
http://www.jrc.ec.europa.eu/

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CERTIFICATION REPORT

Certification of Charpy V-notch reference test pieces of 150 J nominal absorbed energy

Certified Reference Material ERM®-FA415t

A. Lamberty, G. Roebben, T. Linsinger

European Commission, Joint Research Centre
Institute for Reference Materials and Measurements (IRMM), Geel (BE)

Disclaimer

Abstract

This certification report describes the processing and characterisation of ERM®-FA415t, a batch of Charpy V-notch certified reference test pieces. Sets of five of these test pieces are used for the verification of pendulum impact test machines according to EN 10045-2 (Charpy impact test on metallic materials, Part 2. Method for the verification of impact testing machines [1]) or according to ISO 148-2 (Metallic materials - Charpy pendulum impact test – Part 2: Verification of test machines [2]).

The certified value for *KV* (= energy required to break a V-notched test piece using a pendulum impact test machine) is 153 J. The associated uncertainty (9 J, $k = 2$ corresponding to a confidence level of about 95 %) is calculated for the mean of a set of five test pieces.

The absorbed energy (*KV*) is procedurally defined and refers to the impact energy required to break a V-notched bar of standardised dimensions, as defined in EN 10045-1 [3] and ISO 148-1 [4]. The certified value of ERM®-FA415t is traceable to the SI, via the SI-traceable certified value of the master batch ERM®-FA415b, by testing samples of ERM®-FA415b and ERM®-FA415t under repeatability conditions on an impact pendulum verified and calibrated with SI-traceably calibrated tools. The certified value is valid only for strikers with a 2 mm tip radius. The certified value is valid at (20 ± 2) °C.

Contents

Glossary

ASTM	American Society for Testing and Materials
BCR	Community Bureau of Reference
CEN	European Committee for Standardization
CRM	Certified Reference Material
EC	European Commission
EN	European Norm
ERM®	European Reference Material
h	Hour
IMB	International Master Batch
IRMM	Institute for Reference Materials and Measurements
ISO	International Organization for Standardization
JRC	Joint Research Centre
k	Coverage factor
KV	Absorbed energy = energy required to break a V-notched test piece of defined shape and dimensions when tested with a pendulum impact testing machine
KV_{CRM}	Certified KV value of a set of 5 reference test pieces from the Secondary Batch
KV_{MB}	Certified KV value of the Master Batch test pieces
LNE	Laboratoire National de Métrologie et d’Essais
MB	Master Batch
min	Minute
n_{MB}	Number of samples of the Master Batch tested during certification of the Secondary Batch
n_{SB}	Number of samples of the Secondary Batch tested for certification
RSD	Relative standard deviation

s	Standard deviation
SB	Secondary Batch
s_h	Standard deviation of the results of the samples tested to assess the homogeneity of the Secondary Batch
s_{MB}	Standard deviation of the n_{MB} results of the samples of the Master Batch tested for the certification of the Secondary Batch
s_{SB}	Standard deviation of the n_{SB} results of the samples tested for the characterisation of the Secondary Batch
u_{CRM}	Combined standard uncertainty of KV_{CRM}
U_{CRM}	Expanded uncertainty (k = 2, confidence level 95 %) of KV_{CRM}
u_{char}	Standard uncertainty of the result of the characterisation tests
$u_{char,rel}$	Relative standard uncertainty of the result of the characterisation tests
u_h	Standard uncertainty contribution from batch heterogeneity
u_i	Value of uncertainty from contribution i
u_{MB}	Standard uncertainty of KV_{MB}
$u_{MB,rel}$	Relative standard uncertainty of KV_{MB}
% m/m	Mass fraction
$\overline{X}_{MB}$	Mean KV value of the n_{MB} measurements on samples of the Master Batch tested when characterising the Secondary Batch
$\overline{X}_{SB}$	Mean KV value of the n_{SB} results of the samples tested for the characterisation of the Secondary Batch
Δh	difference between the height of the centre of gravity of the pendulum prior to release and at the end of the first half-swing, after breaking the test sample
ν_{eff}	Effective number of degrees of freedom associated with the uncertainty of the certified value

1 Introduction: the Charpy pendulum impact test

The Charpy pendulum impact test is designed to assess the resistance of a material to shock loading. The test, which consists of breaking a notched bar of the test material using a hammer rotating around a fixed horizontal axis, is schematically presented in Figure 1.

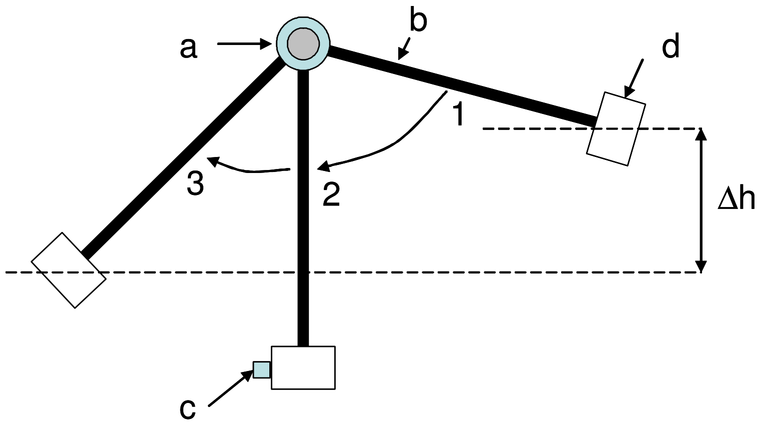

Figure 1: Schematic presentation of the Charpy pendulum impact test, showing a: the horizontal rotation axis of the pendulum, b: the stiff shaft on to which is fixed, d: the hammer, of mass m. The hammer is released from a well-defined height (position 1). The hammer strikes c: the test sample, when the hammer has reached maximum kinetic energy (shaft in vertical position 2). The height reached by the hammer after having broken the sample (position 3) is recorded. The difference in height between position 1 and 3 (Δh) corresponds with a difference in potential energy (= $m \times g \times \Delta h$, with g = gravitation acceleration), and is a measure of the energy required to break the test sample.

The energy absorbed by the test sample depends on the impact pendulum construction and its dynamic behaviour. Methods to verify the performance of an impact pendulum require the use of reference test pieces as described in European, ISO and other international standards [1, 2, 5]. The reference test pieces dealt with in this report comply with a V-notched test piece shape of well-defined geometry [1, 2], schematically shown in Figure 2.

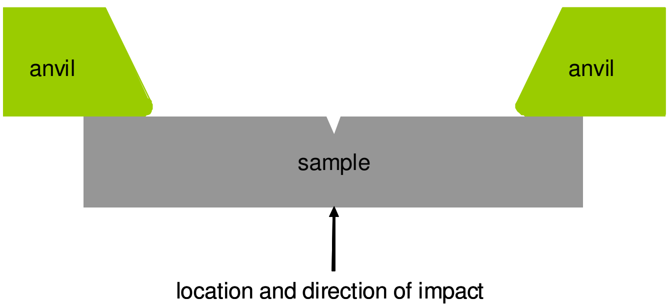

Figure 2: Schematic drawing of a V-notched Charpy sample (top-view), indicating the place and direction of impact.

2 The certification concept of Master Batch and Secondary Batch

2.1 Master and Secondary Batches

The BCR reports by Marchandise et al. [6] and Varma [7] provide details of the certification of BCR "Master Batches" (MB) of Charpy V-notch certified reference test pieces. The certified value of a Master Batch is obtained using an international laboratory intercomparison.

This report describes the production of a "Secondary Batch" (SB) of Charpy V-notch certified reference test pieces at the Institute for Reference Materials and Measurements (IRMM) of the European Commission (EC) Joint Research Centre (JRC). The work was performed in accordance with procedures described in the BCR reports [6] and [7]. The certification of a SB is based on the comparison of a set of SB test pieces with a set of test pieces from the MB with the same nominal energy under repeatability conditions on a single pendulum.

The BCR reports [6] and [7] were published in 1991 and 1999, respectively. Since 2000, the calculation of the certified value and the estimation of its uncertainty have been updated to an approach compliant with the ISO Guide to the Expression of Uncertainty in Measurement [8]. This revised approach was developed and presented by Ingelbrecht et al. [9, 10], and is summarised below.

2.2 Certification of a Secondary Batch of Charpy V-notch test pieces

The certified absorbed energy of a SB of Charpy V-notch reference test pieces (KV_{CRM}) is calculated from the mean KV-value of a set of SB-samples ($\overline{X}_{SB}$) tested on a single pendulum. This value $\overline{X}_{SB}$ has to be corrected for the bias of this particular pendulum. The bias of the pendulum at the moment of testing the samples of the SB, is estimated by comparing the mean KV-value of a number of samples of the MB ($\overline{X}_{MB}$), tested together with the SB samples under repeatability conditions, with the certified value of the MB (KV_{MB}). KV_{CRM} is then calculated as follows [10]:

$$KV_{CRM} = \left[\frac{KV_{MB}}{\overline{X}_{MB}} \cdot \overline{X}_{SB} \right] \qquad \text{Eq. 1}$$

For this approach to be reliable, the pendulum used for the tests on MB and SB in repeatability conditions, must be well performing. In other words, the ratio $\frac{KV_{MB}}{\overline{X}_{MB}}$ must be close to 1. IRMM now allows a difference of 5 % (KV_{MB} > 40 J) or 2 J (KV_{MB} < 40 J) between KV_{MB} and $\overline{x}_{MB}$, corresponding with the level of bias allowed for reference pendulums specified in EN 10045-2 [1] and ISO 148-3 [11].

Also, for reasons of commutability, a comparable response of the pendulum to the MB and SB samples is required. This is the reason why MB and SB samples are made from steel with nominally the same chemical composition, and similar heat treatments. These precautions have to result in a ratio $\frac{KV_{CRM}}{KV_{MB}}$ close to 1. IRMM now allows a difference of 20 % (KV_{MB} > 40 J) or 8 J (KV_{MB} < 40 J) between KV_{CRM} and KV_{MB}.

2.3 Uncertainty of the certified value of a Secondary Batch

The uncertainty of the certified value of the SB is a combination of the uncertainties of the right-hand side factors in Eq. 1. It is clear that the MB-SB approach necessarily results in a larger uncertainty of the certified value of SB in comparison with the MB. The additional uncertainty depends on the uncertainty of the ratio $\overline{X}_{SB} / \overline{X}_{MB}$. The full measurement uncertainty of the values $\overline{X}_{MB}$ and $\overline{X}_{SB}$ is relatively large. However, when all conditions mentioned above (repeatability conditions, pendulum performance, and commutability between Secondary and Master Batch) are fulfilled, then the uncertainties of the values $\overline{X}_{MB}$ and $\overline{X}_{SB}$ have several contributions in common, in particular the uncertainty due to the bias of the pendulum. These shared uncertainty components do not contribute to the uncertainty of the ratio $\overline{X}_{MB} / \overline{X}_{SB}$, and only the standard deviations of the SB and MB results in the MB-SB comparison test need to be taken into account (see also Section 5.3). Thus, the MB-SB comparison approach can produce a value for the uncertainty of KV_{CRM} that is sufficiently small to meet the requirements of the intended use of the certified reference material (CRM).

3 Participants

The processing of the SB (ERM®-FA415t) test pieces was carried out by the Laboratoire National de Métrologie et d'Essais (LNE), Trappes (FR), using steel bars produced at Aubert&Duval, Les Ancizes and Gennevilliers (FR). The MB samples (ERM®-FA415b) used in the characterisation of the SB were provided by IRMM, Geel (BE). The homogeneity of the SB was evaluated based on data obtained at LNE using a pendulum verified according to the criteria imposed by EN 10045-2 [1] and ISO 148-2 [2]. Characterisation of the SB was carried out at IRMM using a pendulum verified according to the criteria imposed by EN 10045-2 [1] and ISO 148-2 [2]. Data evaluation was performed at IRMM.

4 Processing

The ERM®-FA415t test pieces were prepared from ASTM 565 steel. The steel was produced, cast and rolled into bars at Aubert&Duval (see Section 4.1). Production of the test pieces from these bars was performed under the supervision of LNE (see Sections 4.2, 4.3, 4.4 and 4.5).

4.1 Processing of hot-rolled bars

The base material consisted of ASTM 565 XM32 steel, produced at Aubert&Duval. To limit the amount of impurities potentially affecting the homogeneity of the fracture resistance, the following compositional tolerances specified in Table 1 were imposed on the selected steel batch. These tolerances are stricter than generally allowed for ASTM 565.

Table 1: Adapted composition tolerances of ASTM 565 GradeXM32

composition (mass %)						
C	S	P	Si	Mn	Cr	Ni
0.11 - 0.13	< 0.003	<0.018	0.15 – 0.3	0.75 – 0.9	11.25 – 11.65	2.55 – 2.75
Mo	Cu	Al	V	W	N	
1.55 – 1.7	< 0.2	< 0.01	0.25 – 0.3	< 0.1	0.025 - 0.04	

The ingot (block of steel resulting from the casting process) was hot rolled, resulting in bars that were 6.4 m long and with a squared cross-section of 12 mm. For the ERM®-FA415t batch, steel was used from ingot number SC2252W.

4.2 Heat treatment of hot-rolled bars

The heat treatment of the hot-rolled bars was performed at Aubert&Duval, Gennevilliers (FR). 15 bars were heat-treated together. Bars were placed onto rollers which slowly move the bars back and forth inside the furnace during the heat treatment to increase the homogeneity of the resulting microstructure. The first heat treatment was an austenisation treatment performed in a furnace of 'class 10 °C' [1] at 980 °C for 30 min. From this furnace, the bars were quenched into oil at 40 °C. After the oil-quench, the samples were annealed in a furnace of 'class 5 °C' at 745 °C for 300 min. After this annealing treatment, the samples were cooled down in air.

4.3 Machining of Charpy test pieces

After heat treatment, a limited number of samples (5) were machined for a preliminary check of the obtained energy level. Results indicated an average *KV*-level (154.8 J) within the desired energy range (145 J to 155 J).
The samples were machined to final dimensions. The most important dimensions (sides, depth of notch, notch-tip radius) of 30 samples were checked against the contractual criteria deduced from ISO 148-3 [11] and EN 10045-2 [1] before they were impact tested on the Tinius Olsen pendulum at LNE.

4.4 Quality control

When all samples from the batch were fully machined, a random selection of 30 samples was made. The dimensions of the 30 samples were checked on January 23, 2007 against criteria specified in ISO 148-3 [11] (height 10.00 $\pm$ 0.06 mm, width 10.00 ± 0.07 mm, depth of notch 2.00 ± 0.06 mm, radius at notch root 0.250 ± 0.025 mm). All samples met all requirements with the exception of one sample for which the half-length was slightly outside the specifications.

The 30 samples checked for geometrical compliance were impact tested on January 25, 2007 (laboratory temperature 20 °C, relative humidity 47 %). The results are reported in certificate LNE n° E090227/CQPE/4 [12]. The average *KV* of the 30 samples of batch FA415t was 151.7 J (s = 5.6 J, RSD = 3.7 %), which is within the desired energy range (145 J to 155 J). The standard deviation of the test results (*s* =

[1] In a furnace of 'class x °C', the variation of the temperature is smaller than x °C. The furnaces used have 10 heating zones. Each zone has 3 controlling thermocouples and 3 measurement thermocouples. These are regularly calibrated. When one faulty thermocouple is detected, it is replaced by a thermocouple produced with wire from the same roll. When a roll is exhausted, all thermocouples are replaced with new ones.

5.6 J, *RSD* = 3.7 %) was slightly higher than the maximum allowed 3 %. The variation was checked again during the certification tests at IRMM (see Section 5).

4.5 Packaging and storage

Finally, the samples were cleaned and packed in sets of 5, in oil-filled and closed plastic bags. These oil-filled bags, together with a label, again were packed in a sealed plastic bag, and shipped to IRMM. After arrival (February 2007), the 1400 samples (280 sets) were registered and stored at room temperature, pending distribution.

5 Characterisation

5.1 Characterisation tests

A random selection of 20 samples from the full ERM®-FA415t batch (sets 1, 57, 114, and 218, each set consisting of a random selection of test pieces from the whole batch) were tested under repeatability conditions with 30 samples from MB ERM®-FA415b (sets 38, 42, 46, 68, 72 and 80), using the Instron Wolpert PW 30 (serial number 7300 H1527) machine of IRMM, an impact pendulum yearly verified according to procedures described in EN 10045-2 [1] and ISO 148-2 [2]. Tests were performed on February 24, and 25, 2009 (laboratory temperature 20 ± 1 °C), in accordance with EN 10045-1 [3] and ISO 148-1 [4]. The measured absorbed energy values were corrected for friction and windage losses.

The data obtained on individual test pieces are shown in Figure 3 and in Annex 1. In Figure 3 linear trend-lines are added to illustrate the absence of a significant trend in the results with the analysis sequence. The results of the measurements are summarised in Table 2.

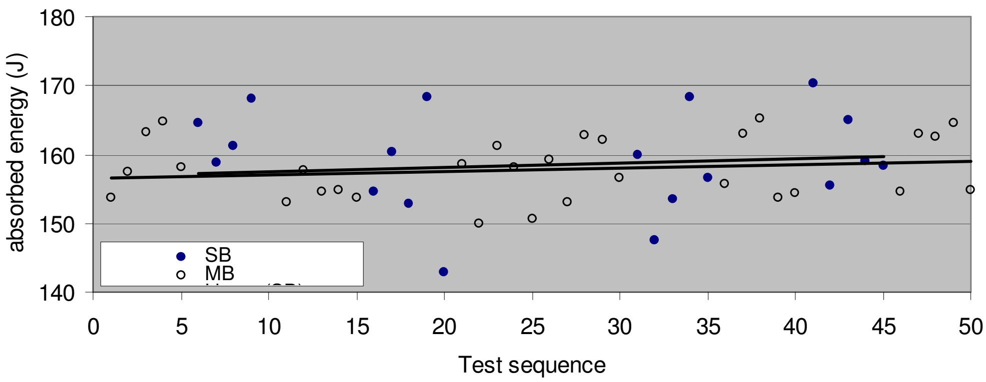

Figure 3: Absorbed energy values of the 20 test pieces of ERM®-FA415t and 30 test pieces of ERM®-FA415b displayed in the actual test sequence.

Table 2: Characterisation measurements of Batch ERM®-FA415t.

	Number of test pieces	Mean value	Standard deviation	Relative standard deviation
	n_{MB} or n_{SB}	$\overline{X}_{MB}$, $\overline{X}_{SB}$ (J)	s_{MB} , s_{SB} (J)	*RSD* (%)
ERM®-FA415b (MB)	30	157.82	4.45	2.82
ERM®-FA415t (SB)	20	158.39	8.01	5.06

The relative standard deviation of the 20 SB-results (5.06 %) is after rounding within the EN 10045-2 and ISO 148-3 acceptance criteria for a batch of reference materials (RSD ≤ 5 %). In addition, the difference between $\overline{X}_{MB}$ and $\overline{X}_{SB}$ is smaller than 20 %, the level used to assess the similarity of Master Batch and Secondary Batch behaviour (see 2.2).

5.2 Data from Master Batch ERM®-FA415b

To calculate KV_{CRM} for ERM®-FA415t one needs KV_{MB} of the MB used, i.e. EM®-FA415b.
Table 3 shows the main MB-data, taken from the Certificate of Analysis of ERM®-FA415b (Annex 2), which is the revised, ERM-version of the originally issued certificate, based on the certification report of the MB [7]. The certified value was obtained from an interlaboratory comparison with 9 laboratories.

Table 3: Data from the certification of Master Batch ERM®-FA415b [6].

	Certified absorbed energy of Master Batch	Standard uncertainty of KV_{MB}	Standard uncertainty of KV_{MB}
	KV_{MB} (J)	u_{MB} (J)	u_{MB} (%)
ERM®-FA415b	152.4	1.1	0.7

The values KV_{MB} (Table 3) and $\overline{X}_{MB}$ () are less than 5 % different, confirming that the pendulum used for the characterisation of the secondary batch is functioning with a sufficiently low bias (see 2.2).

5.3 Calculation of KV_{CRM} and of u_{char}

From the data in and Table 3, and using Eq. 1, one readily obtains that KV_{CRM} = 153 J (rounding in accordance with uncertainty; see 6). The uncertainty associated with the characterisation of the SB, u_{char}, is assessed as in Eq. 2 [10], which sums the relative uncertainties of the three factors appearing in Eq. 1:

$$u_{char} = KV_{CRM}\sqrt{\frac{{u_{MB}}^2}{KV_{MB}^2} + \frac{s_{SB}^2}{n_{SB}\cdot\overline{X}_{SB}^2} + \frac{s_{MB}^2}{n_{MB}\cdot\overline{X}_{MB}^2}} \qquad \text{Eq. 2}$$

$\overline{X}_{SB}$ and $\overline{X}_{MB}$ were obtained under repeatability conditions. Therefore, the uncertainty of the ratio $\overline{X}_{SB} / \overline{X}_{MB}$ is not affected by the contributions from reproducibility and bias of the pendulum used to compare MB and SB.

Table 4 summarises the input quantities of the u_{char} uncertainty budget, their respective statistical properties, and shows how they were combined. The effective number of degrees of freedom for u_{char} is obtained using the Welch-Satterthwaite equation [8].

Table 4: Uncertainty budget for u_{char}

symbol	measured value (J)	source of uncertainty	standard uncertainty value (J)	probability distribution	divisor[1]	sensitivity coefficient[2]	relative standard uncertainty (%)	degrees of freedom
KV_{MB}	152.4	certification of MB	1.1	normal	1	1	0.72	8
$\overline{X}_{SB}$	158.39	comparison of SB and MB in repeatability conditions	1.79	normal	1	1	1.13	19
$\overline{X}_{MB}$	157.82		0.81	normal	1	1	0.52	29
						u_{char} (%)	1.44	34
						u_{char} (J)	2.20	

6 Homogeneity

The test pieces constituting a CRM unit are sampled from the SB, which is sufficiently homogeneous (*RSD* = 5.06 %), but not perfectly homogeneous. Therefore, as for most reference materials, a separate homogeneity contribution u_h to the uncertainty of the certified value is required.

Here, u_h is estimated from s_{SB}, the standard deviation of the results shown in Table 2. As is required for a homogeneity test, the samples were randomly selected from the whole batch. The number of samples tested (20) is sufficient to reflect the homogeneity of the full SB (1400 samples).

The effect of s_{SB} on the uncertainty of the certified value depends on the number of samples over which the KV-value is averaged. EN 10045-2 [1] and in ISO 148-2 [2] specify that pendulum 'indirect verification' with CRMs must be performed using 5 test pieces. Therefore, a CRM-unit consists of 5 test pieces, and $u_h = \frac{s_{SB}}{\sqrt{5}} = 3.58\ \text{J}$. u_h is probably a slight overestimation, since it contains also the repeatability of the instrument. However, the latter cannot be separated or separately measured.

[1] Divisor: number used to calculate standard uncertainty from non-standard-uncertainty expression of uncertainty (e.g.: coverage factor to adapt expanded uncertainty to standard uncertainty, or factor to transform bounds of rectangular distribution into standard uncertainty of equivalent normal distribution).

[2] Sensitivity coefficient: used to multiply an input quantity to express it in terms of the output quantity.

7 Stability

Microstructural stability of the certified reference test pieces is obtained by the annealing treatment to which the samples were subjected after the austenisation treatment. Annealing is performed at temperatures where the equilibrium phases are the same as the (meta-)stable phases at ambient temperature (α-Fe and Fe_3C). The only driving force for instability stems from the difference in solubility of interstitial elements in the α-Fe matrix, between annealing and ambient temperature. Relaxation of residual (micro-)stress by short-range diffusion or the additional formation or growth of precipitates during the shelf-life of the certified reference test pieces is expected to proceed but slowly. Given the large sample-to-sample heterogeneity, the ageing effects are undetectable when testing limited numbers of samples, and the uncertainty contribution from instability is considered to be insignificant. Until further notice, it is decided to specify a limited shelf-life. A period of 10 years is chosen, counting from the date of the characterisation tests on the SB. Since batch ERM®-FA415t was characterised in February, 2009, the validity of the certificate stretches until February, 2019. This validity may be extended as further evidence of stability becomes available.

Rather than neglecting the stability issue, efforts are spent to better establish the stability of the certified values of batches of Charpy CRMs. The stability of the absorbed energy of Charpy V-notch certified reference test pieces was first systematically investigated for samples of nominally 120 J by Pauwels et al., who did not observe measurable changes of absorbed energy [13]. New evidence for the stability of the reference test pieces produced from AISI 4340 steel of other energy levels (nominally 15 J, 30 J and 100 J) has been obtained recently, during the International Master Batch (IMB) project [14]. In the IMB-project, the stability of the certified test pieces is confirmed by the unchanged value of the mean of means of the absorbed energy obtained on 7 reference pendulums over a three year period.

8 Calculation of certified value, combined and expanded uncertainty

As shown in 5.3 KV_{CRM} = 153 J. The uncertainty of the certified value is obtained by combining the contributions from the characterisation study, u_{char}, and from the homogeneity assessment, u_h. The absolute values of these contributions are quadratically summed, and the approach is summarized in the uncertainty budget shown in Table 5.

Table 5: Uncertainty budget of KV_{CRM}

symbol	source of uncertainty	absolute value (J)	divisor[1]	sensitivity coefficient[2]	u_i (J)	degrees of freedom
u_{char}	characterisation of SB	2.20	1	1	2.20	34
u_h	homogeneity of SB	3.58	1	1	3.58	19
Combined standard uncertainty, u_{CRM}					4.20	33
Expanded Uncertainty, k = 2, U_{CRM}					8.40	

The relevant number of degrees of freedom calculated using the Welch-Satterthwaite equation [8], is sufficiently large (ν_{eff} = 33) to justify the use of a coverage factor k = 2 to expand the confidence level to about 95 %. The obtained expanded uncertainty provides justification for the SB-MB approach followed: U_{CRM} is sufficiently smaller (5.6 %) than the verification criterion of 10 % (for industrial pendulums [1, 2]).

9 Metrological traceability

The certified property is defined by the Charpy pendulum impact test procedure described in EN 10045-1 [3] and ISO 148-1 [4].

The certified value of the MB ERM®-FA415b is traceable to the SI, since it was obtained using an interlaboratory comparison, involving a representative selection of qualified laboratories performing the tests in accordance with the standard procedures and using instruments verified and calibrated with SI-traceable calibration tools.

The certified value of ERM®-FA415t is made traceable to the SI-traceable certified value of the MB by testing SB and MB samples in repeatability conditions on an impact pendulum verified and calibrated with SI-traceably calibrated tools. Therefore, the certified value of ERM®-FA415t is traceable to the International System of Units (SI) via the corresponding Master Batch ERM®-FA415b of the same nominal absorbed energy (150 J). Absorbed energy *KV* is a method-specific value, and can only be obtained by following the procedures specified in EN 10045-1 [3] and ISO 148-1 [4].

[1] Divisor: number used to calculate standard uncertainty from non-standard-uncertainty expression of uncertainty (e.g.: coverage factor to adapt expanded uncertainty to standard uncertainty, or factor to transform bounds of rectangular distribution into standard uncertainty of equivalent normal distribution).

[2] Sensitivity coefficient: used to multiply an input quantity to express it in terms of the output quantity.

10 Commutability

The intended use of the certified reference test pieces is the verification of Charpy impact pendulums. During the certification of the MB, different pendulums were used, each equipped with an ISO-type striker of 2 mm tip radius. Until further notice, the certified values are not to be used when the test pieces are broken with an ASTM-type striker of 8 mm tip radius.

11 Summary of results

The certified value and associated uncertainty are summarized in Table 6.

Table 6: Certified value and associated uncertainties for ERM®-FA415t.

Steel Charpy V-notch test pieces		
	Certified value [2)] (J)	Uncertainty [3)] (J)
Absorbed energy (*KV*) [1)]	153	9

1) The absorbed energy (*KV*) is a method defined measurand. KV is the impact energy required to break a V-notched bar of standardised dimensions, as defined in EN 10045-1 and ISO 148-1. The certified value is valid only for strikers with a 2 mm tip radius, and in the temperature range of (20 ± 2) °C.

2) The certified value of ERM®-FA415t, and its uncertainty, are traceable to the International System of Units (SI), via the master batch ERM®-FA415b of the same nominal absorbed energy (150 J) by testing samples of ERM®-FA415b and ERM®-FA415t under repeatability conditions on an impact pendulum verified and calibrated with SI-traceably calibrated tools.

3) Estimated expanded uncertainty of the mean *KV* of the 5 specimens (delivered as 1 set), with a coverage factor $k = 2$, corresponding to a level of confidence of about 95 %, as defined in the ISO/IEC Guide 98-3:2008; Uncertainty of measurement – Part 3: Guide to the expression of uncertainty in measurement (GUM). The number of degrees of freedom of the certified uncertainty, $\nu_{eff} = 33$.

12 Instructions for use

12.1 Intended use

Samples of ERM®-FA415t correspond with the '(certified) BCR test pieces' as referred to in EN 10045-2 [1], as well as with the 'certified reference test pieces' as defined in ISO 148-3 [11] (albeit with testing 20 rather than 25 test pieces). Sets of five of these certified reference test pieces are intended for the indirect verification of impact testing machines with a striker of 2 mm tip radius according to procedures described in detail in EN 10045-2 [1] and ISO 148-2 [2]. The indirect verification provides a punctual assessment of the bias of the user's Charpy pendulum impact machine.

12.2 Sample preparation

Special attention is drawn to the cleaning and conditioning of the specimens prior to testing. It is mandatory to remove the oil from the sample surface prior to testing, without damaging the edges of the sample. Between the moment of removing the protective oil layer and the actual test, corrosion can occur. This must be avoided by limiting this period of time, while keeping the sample clean.

The following procedure is considered good practice.

1. First use absorbent cleaning-tissue to remove the excess oil. Pay particular attention to the notch of the sample, but do not use hard (e.g. steel) brushes to remove the oil from the notch.
2. Submerge the samples in ethanol for about 5 minutes. Use of ultrasonication is encouraged, but only if the edges of the samples are prevented from rubbing against each other. To reduce the consumption of solvent, it is allowed to make a first cleaning step with detergent, immediately prior to the solvent step.
3. Once samples are removed from the solvent, only manipulate the samples wearing clean gloves. This is to prevent development of corrosion between the time of cleaning and the actual test.
4. Before testing, bring the specimens to the test temperature (20 ± 2 °C). To assure thermal equilibrium is reached, move the specimens to the test laboratory at least 3 hours before the tests.

12.3 Pendulum impact tests

After cleaning, the 5 samples constituting a CRM-unit need to be broken with a pendulum impact test machine in accordance with EN 10045-2 [1] or ISO 148-2 [2] standards. Prior to the tests, the anvils must be cleaned. It must be noted that Charpy test pieces sometimes leave debris on the Charpy pendulum anvils. Therefore, the anvils must be checked regularly and if debris is found, it must be removed.

The comparison of the indirect verification results with the certified value and uncertainty must be based on the mean of the 5 measured *KV* values, because the calculation of the uncertainty of the certified value is based on this sample size.

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Annex 1

Results of characterisation measurements of ERM®-FA415t as measured according to EN 10045-1 and ISO 148-1 at IRMM, February 2009.

	Master Batch **ERM®-FA415b**	**Secondary Batch** **ERM®-FA415t**
	***KV* (J)**	***KV* (J)**
1	153.70	164.46
2	157.43	158.80
3	163.10	161.13
4	164.66	168.10
5	158.21	142.30
6	152.98	154.68
7	157.62	160.42
8	154.49	152.92
9	154.88	168.36
10	153.70	142.96
11	158.66	159.84
12	149.97	147.49
13	161.20	153.57
14	158.21	168.23
15	150.63	156.51
16	159.25	170.17
17	152.98	155.40
18	162.78	164.92
19	162.19	159.06
20	156.64	158.41
21	155.73	
22	162.97	
23	165.12	
24	153.77	
25	154.36	
26	154.54	
27	162.97	
28	162.58	
29	164.53	
30	154.75	
Mean (J)	**157.82**	**158.39**
Standard deviation (J)	**4.45**	**8.01**
***RSD* (%)**	**2.8**	**5.1**

Annex 2

CERTIFICATE OF ANALYSIS

ERM®- FA415b

Steel Charpy V-notch test pieces (nominal absorbed energy[1)] 150 J, Master Batch)		
Parameter	Certified value [2)] (J)	Uncertainty [3)] (J)
Absorbed energy (KV) at 20 ± 2 °C, according to EN 10045-1 and ISO 148	152.4	1.1

1) The term absorbed energy is defined in EN 10045-1 and ISO 148 and refers to the impact energy required to break a V-notched bar of standardised dimensions.

2) Mean absorbed energy of test pieces from batch ERM®-FA415b. The certified value was obtained as the mean of means of absorbed energies measured at 9 laboratories. At each laboratory, 10 test pieces were broken. The certified value is traceable to the Charpy impact test method as described in EN 10045-1 and ISO 148. The certified value is valid only for impact hammers with a 2 mm striker tip radius.

3) Half-width of the 68 % confidence interval of the mean absorbed energy defined in 2), estimated as $\frac{\sigma_m}{\sqrt{9}}$, with σ_m the standard deviation of the mean of the mean values obtained at the 9 participating laboratories.

This certificate is valid until October 2009; this validity may be extended as further evidence of stability becomes available.

NOTE

European Reference Material ERM®-FA415b was originally certified as BCR-415 B. It was produced and certified under the responsibility of IRMM according to the principles laid down in the technical guidelines of the European Reference Materials® co-operation agreement between BAM-IRMM-LGC. Information on these guidelines is available on the Internet (http://www.erm-crm.org).

Accepted as an ERM®, Geel, July 2005

Signed: ____________

Prof. Dr. Hendrik Emons
Unit for Reference Materials
EC - JRC - IRMM
Retieseweg 111
2440 Geel, Belgium

All following pages are an integral part of the certificate.

DESCRIPTION OF THE SAMPLE

A unit consists of five Charpy V-notch test pieces, which are rectangular steel bars of nominal dimensions 55 mm x 10 mm x 10 mm, with one V-notch, accurately machined to tolerances imposed in EN 10045-1 and ISO 148. The five specimens are packed together in a plastic bag filled with oil to prevent oxidation.

INSTRUCTIONS FOR USE

The ERM-FA415b batch is intended to be used as a 'Master Batch'. Master Batch test pieces are not for sale. They are used to traceably certify Secondary Batches of Charpy V-notch reference test pieces of the same type of steel with the same nominal absorbed energy (here 150 J).

To characterise a secondary batch, a selection of samples from the secondary batch have to be broken under repeatability conditions together with Master Batch test pieces. The certified value of the Master Batch and its associated uncertainty are used in the calculation of the certified value and its combined and expanded uncertainty of a set of 5 specimens from a Secondary Batch. Sets of 5 samples of Secondary Batches are distributed as certified reference test pieces for the verification of Charpy impact test machines in accordance with EN 10045-2 and ISO 148-2.

Special attention is drawn to cleaning and conditioning of the specimens prior to testing. The following procedure is recommended:

1. Wipe excess oil from the specimens with cellulose paper.

2. Immerse the specimens in a clean bath of degreasing solvent for about 5 min.

3. Wipe the specimens with cellulose paper and let dry.

4. Before testing, bring the specimens to the test temperature (20 ± 2 °C). To assure thermal equilibrium is reached, move the specimens to the test laboratory at least 12 h before the tests.

After cleaning, the user must avoid touching the specimens with the fingers (wear clean gloves). Vigorous cleaning methods affecting the roughness of the specimen surface or possibly causing deformation or indentation of the specimen edges should be avoided, as this can result in obtaining erroneous data.

The cleaned samples need to be broken with an impact pendulum in accordance with EN 10045-1 or ISO 148 standards.

Unlike Charpy test pieces of lower nominal impact energies, samples from ERM-FA415 batches sometimes leave debris on the Charpy pendulum anvils. Therefore, after each impact, the anvils must be checked and if debris is found, it must be removed.

After testing, the user is recommended to inspect the traces/imprints left behind by the anvils and hammer on the two halves of the broken specimen. Asymmetry of these marks can indicate problems with the machine geometry or the positioning of the sample prior to impact. If so desired, broken samples can be stored for later inspection of the anvil and striker marks.

STORAGE

Specimens should be kept at ambient temperature in their original packing until used. The European Commission cannot be held responsible for changes that happen during storage of the material at the customer's premises, especially of opened samples.

SAFETY INFORMATION

Precautions need to be taken to avoid injury of the operator by broken specimens when operating the Charpy impact pendulum.

METHOD USED FOR CERTIFICATION

Charpy pendulum impact tests in accordance with EN 10045-1 and ISO 148, using pendulum impact machines with a 2 mm striker tip radius.

PARTICIPANTS

- Ansaldo Energia S.p.A., Legnano (IT)
- Bundesanstalt für Materialforschung und –prüfung (BAM), Berlin (DE)
- Cogne Acciai Speciali srl, Aosta (IT)
- Inco Test, Hereford (UK)
- Instron Ltd., High Wycombe (UK)
- Instituto de Soldadura e Qualidade (ISQ), Oeiras (PT)
- Laboratoire National d'Essais (LNE), Trappes (FR)
- Lloyd's Register, Croydon (UK)
- National Institute of Standards and Technology (NIST), Boulder, CO (US)
- National Physical Laboratory (NPL), Teddington (UK)
- Rolls Royce Ltd., Derby (UK)
- Studiecentrum voor Kernenergie (SCK•CEN), Mol (BE)
- Universidad Carlos III, Madrid (ES)

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NOTE

A detailed technical report of the Master Batch certification project ("The certification of two new Master Batches of V-notch Charpy impact toughness specimens in accordance with EN 10045-2: 1992", R. K. Varma, bcr information, EUR 18947 EN, 1999) can be obtained from IRMM on explicit request. In this report, the ERM-FA415b batch is called "CRM 415 B".

European Commission – Joint Research Centre
Institute for Reference Materials and Measurements (IRMM)
Retieseweg 111, B - 2440 Geel (Belgium)
Telephone: +32-14-571.722 - Telefax: +32-14-590.406

European Commission

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Abstract

This certification report describes the processing and characterisation of ERM®-FA415t, a batch of Charpy V-notch certified reference test pieces. Sets of five of these test pieces are used for the verification of pendulum impact test machines according to EN 10045-2 (Charpy impact test on metallic materials, Part 2. Method for the verification of impact testing machines [1]) or according to ISO 148-2 (Metallic materials - Charpy pendulum impact test – Part 2: Verification of test machines [2]).

The certified value for *KV* (= energy required to break a V-notched test piece using a pendulum impact test machine) is 153 J. The associated uncertainty (9 J, k = 2 corresponding to a confidence level of about 95 %) is calculated for the mean of a set of five test pieces.

The absorbed energy (*KV*) is procedurally defined and refers to the impact energy required to break a V-notched bar of standardised dimensions, as defined in EN 10045-1 [3] and ISO 148-1 [4]. The certified value of ERM®-FA415t is traceable to the SI, via the SI-traceable certified value of the master batch ERM®-FA415b, by testing samples of ERM®-FA415b and ERM®-FA415t under repeatability conditions on an impact pendulum verified and calibrated with SI-traceably calibrated tools. The certified value is valid only for strikers with a 2 mm tip radius. The certified value is valid at (20 ± 2) °C.

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